Notebook Series -- Notebooks by Experimenters Other Than Edison
Battery Experiments -- Experimenters' Books
H. H. Smith Notebook, N-13-08-02.1

This notebook was used by Harold H. Smith during August 1913, April-June 1915, and December 1916 for experimental notes on nickel flake, hydrate, and other battery components. Most of the entries deal with the daily control of flake, which generally varied between four and six ounces per cell. Some of the entries mention Edison's attention to the flake reports and his interest in the amount of flake in the cells. A note from May 6, 1915, indicates that initially Edison "rigidly insisted upon flake not less than 4 mg per dump" but that the low limit was subsequently "reduced over [Walter N.] Archer's protest to 3.4 mg." Also included is an entry regarding a "special test" that was performed on January 3, 1911. The notes indicate that Robert A. Bachman, Thomas D. Greenley, Ademor N. Petit, and employees named McClain and Reed were also involved in the work. The spine is labeled "Report." The pages are unnumbered, and the book has been used in both directions. Approximately 50 pages have been used.

13.08.02

H H Smith

46288

Pome Co.,

MFG. STATIONERS,
69 FULTON STREET,
NEW YORK

Daily control of flake began 8/2/13

~~In two weeks~~ Previously flake varied from 4 to 6 ounces per cell

Previous to this the fl. weights were high

Perfs. change 12/10/14 to .010" x .015" (smaller than before)

When loading weight of flake has been high that of hydrate has been low.

1187	5080.
1131	5079.
1155	5043
1151	5065
1108	5069

Greenley makes statement that in sediments the proportions of constituents are about constant regardless of amount of sediment, namely

Ni 45 - 47 %
Fe 12 - 15 %

This naturally suggests that sediment is not active material.

Sometimes tubes are not soft in cells having excess sediment. This would also suggest that sediment is not active material.

<u>Question</u> Is it chemically possible that solution dissolves nickel and iron from grids, tubes, or active material or cases plates and precipitates it under influence of current or otherwise?

<u>Ans.</u> Yes by small amounts of acid radicals.

The fact that the trouble did not always exists argues against this point.

In time of little trouble from sediment (records kept only from 1911, on) the loading weight of flake was high and that of hydrate low

2.5 lbs. hydrate 1130 grms.
5.7 oz. flake 161 "

$$\frac{1130}{161} = \frac{32.6}{4}$$

At times (May & June 1911) the ratio was as low as:

2.6 lbs. hydrate 1090 grms.
7.7 oz. flake 218 "

$$\frac{1090}{218} = \frac{20}{4}$$

Research Dept. tubes are all loaded at ratio of

$$\frac{24}{4}$$

At times they have been at

$$\frac{27}{4}$$

(over)

Mr Clain suggests that the high ratio has some relation to sediment troubles, which seems plausible according to the following course of reasoning:

Flake is springy (it must be curly - flat flake doesn't do) - when you grasp a handful of it, it is compressible and resilient like a handful of feathers or silk.

When tamped it must be somewhat springy after the pressure is removed.

The active material (hydrate) tends to swell in operation of cell.

If flake is springy it will take up expansion of hydrate.

If flake is not springy it will not take up expansion and tendency will be for material to squeeze out through perforations, or burst the tube.

(WEN)

If layer of flake is thin it obviously will not have same springiness as when comparatively thick.

Therefore it would seem that the ratio of hydrate to flake should be low.

Experiment Load tubes with large perforations and different ratios of hydrate to flake. Run regular (including high temp.) and at high rates. Note capacity and sediment.

Chemical

Old hydrate was bright green and clean cut (sharp particles).

Present hydrate is dull green and dark gray. Soft — not clean cut particles. Dead looking.

Greenly points out that chlorine used in mfr. may make dark color. Also temp. of drying has an effect. Showed that Cu hydrate was dark + dead looking when produced hot — bright looking when produced cold.

He also demonstrated effect of Br (which he says is similar to that of Cl) on bright hydrate (#1256). Caused it to get dark + grayish very much like present hydrate.

Mellor says dark color

is due to Co but samples he has made up without Co show same general color as that made with Co.

Soft consistancy of present hydrate is shown: ~~by [illegible]~~

1. By sound in crushing rolls.
2. By producing effect in loading machines.
3. By comparison of loaded pencils with tube stock removed.

Why are some sediments loose and shiny, while others are hard and compact?

Why does high temp. during change compact sediment?

What is effect of loading 3 dumps per stamp on sediment?

Perhaps layers are not distinct and therefore cushioning effect of flake is lost.

Also effect of flake closing perforations may be partly lost.

19606	3 dumps.	61.3 grams
19879	2 "	46.2 "

	19606	19879
K_2SO_4	.213	.887
Ni	32.8 %	34.5 %
Fe	14.6 "	14.9 "

$\frac{61.3}{46.2} = 133\ \%$

What is effect of stronger Renewal solution

Cells cleaned out and rerun will not reprecipitate large amount of sediment. Greenly reports cases of one year's standing after cells cleaned.

Case of L.V. cells opened a year ago and again recently — no sediment when opened second time.

Small amounts of acid radicals in electrolyte will attack the active material and precipitate as sediment when current ceases. Radical is then released and again performs the operation.

This may account for a slight, natural, progressive sediment which is natural and unavoidable.

Not important, probably, in this investigation except where comparatively large amounts of impurity exist.

These acids will attack both active materials & grids but active materials are more susceptible than metals.

With present hydrate, the loading of tubes fills up space beside high seam with fine green powder instead of stretching tube stock to fill out ~~such~~ this space

Samples of filler from old and new tubes produced by Lystrup. ~~Old~~ Manson from old tubes could not be readily crushed in the fingers, and the entire pencil could be removed intact. The pencil in new tubes would break in half-a-dozen or more places, and the glass would crumble under the lightest pressure.

In the old tubes whole particles as originally loaded could be separated — in new ones there was only a very fine powder.

Old load's was very regular and "cup effect" of flake was plainly evident.

(over)

Mixing takes place in later tubes — lines not distinct.

The dump of hydrate is finely powdered and is agitated by the tamping rod at the next tamp. The rod acts as a piston forcing the air before it. The air is blown out through the perforations and up outside the tube. This probably blows up a cloud of powdered hydrate and flake and obliterates distinct lines.

Outside of old pencil — much flake appears.

Outside of new pencil — more hydrate appears than flake.

Old tube, when split, could be polished. Present tubes will not polish.

Sediment determinations
weight in grams.

After 16 Runs: Per cell	Total 2 cells 1st low-mate hot 2nd high " "	Total 4 cells	After 59 Runs		
.1014	.3391 (2581)		.0167	.0530	
.2377		0.7390	.0362		.1635
.2432	.3999 (4309)		.0555	.1105	
.1567		30.5	.0550		
.1570	.3565		.0276	.0473	
.1995		1.0091	.0177		.1218
.3927	.6526		.0428	.0745	
.2599		41.7	.0317		
.2002	.5019		.0003	.0110	
.3017		1.1703	.0107		.1355
.3587	.6684		.0638	.1245	
.3097		48.3	.0607		
.3007	.6454		.0126	.0249	
.3447		1.3773	.0123		.1494
.3487	.7319		.0616	.1245	
.3832		56.8	.0629		
.3227	.7534		.0187	.0276	
.4307		1.6244	.0089		.1776
.3655	.8710		.0806	.1500	
.4855		67.0	.0694		
.1305	.2660		.0014	.0215	
.1355		0.7459	.0201		.0969
.2332	.4799		.0468	.0754	
.2467		30.9	.0286		

Hydrate 1256
Loading Experiment

Mg. per dump. Hyd.	Flake	Loading weight	Average	No. of Dumps	Average	Mg. per dump.
20	4	7.515		.324		
		7.495	7.524	322	325.5	23.2
16 2/3 % Flake		7.530		329		
		7.575		327		
24	4	7.380		268		
		7.390		269		
14.3 % Flake		7.385	7.375	263	266.75	27.6
		7.365		267		
27	4	7.240		228		
		7.210		227		
12.9 % Flake		7.255	7.235	226	228.5	31.6
		7.235		233		
30	4	7.200		209		
		7.175		211		
11.8 % Flake		7.175	7.176	206	208.75	34.4
		7.155		207		
34	4	7.100		193		
		7.085		195		
10.5 % Flake		7.120	7.101	197	195.25	36.4
		7.100		196		
15	3	7.850		441		
		7.855	7.843	441	448.75	17.5
16 2/3 % Flake		7.825		461		
		7.840		452		

.3907	.6994		.0273	.0653	
.3087		1.4203	.0360		.1399
.2512			.0491	.0746	
.4697	.7209	58.6	.0255		
2.5917	6.6944		.1942	1.8207	
4.1027		15.8978	1.6465		4.0617
4.9112	9.2034		.8464	2.2210	
4.2922		656	1.3746		

1256

Mg. per dump. Hyd.	Flake	Loading weight	Average	No. of Dumps	Average	Mg per dump
27	3	7.205		235		
		7.220		239		
10% Flake		7.220	7.2125	239	238.25	30.27
		7.205		240		
30	0	6.735		216		
		6.740		221		
		6.710	6.730	220	218.75	30.75
		6.735		218		

	1st Run Averages			3rd Run-Aver.		
20/4	1271	.1848 gm	7.524	1237	325.5	23.2
24/4	1274	.2523	7.375	1259	266.7	27.6
27/4	1273	.2926	7.235	1231	228.5	31.6
30/4	1283	.3443	7.176	1220	208.7	34.4
34/4	1253	.4061	7.101	1190	195.2	36.4
15/3	1215	.1865	7.843	1203	448.7	17.5
27/3	1269	.3551	7.213	1237	238.2	30.27
30/0	31	3.9744	6.730	70	218.7	30.75

Weight & number of dumps seem to be largely determined by weight of hydrate per dump and to be practically independent of weight of flake per dump, within limits.

Loading weights as indicated by daily & weekly reports from Tube Loading Dept are higher than actual as they include sweepings or waste.

Sweepings per mould (6 tubes) average about 2.650 grams including both flake and hydrate.

A badly burst tube cut open showed very thin layers of flake & comparatively thick layers of hydrate

Sediment

After 16 Runs: Per cell	Total 2 Cells 1st. low rate 1st. 2nd high " "	Total 4 cells	After 59 Runs		
.0710	.1715	.4960	.0137	.0150	.0835
.1005			.0013		
.2110	.3245		.0548	.0685	
.1135			.0117		
.2135	.4098	.7483	.0029	.0047	.1006
.1963			.0018		
.2570	.3385		.0581	.0959	
.0815			.0378		
.2000	.4630	1.0665	.0096	.0204	.1576
.2630			.0108		
.3335	.6035		.0615	.1372	
.2700			.0757		
.3125	.5475	1.1520	.0157	.0313	.1432
.2350			.0156		
.2840	.6045		.0713	.1019	
.3205			.0306		
.2900	.5800	1.2907	.0129	.0379	.2035
Runner 3/100 .0113			.0250		
.3565	.7107		.0734	.1656	
.3542			.0922		
.1300	.3040	.6720	.0612	.0635	.1509
.0740			.0023		
.1670	.3680		.0417	.0874	
.2010.			.0457		

Hydrate 102 Special

5782 - 5788 - 5794 - 5795 - 5800.

Mg. per dump Hyd.	Flake	Loading weight	Average	No. of Dumps	Average	Mg. per Dump.
20	4	7.725	7.711	319	322.0	24
		7.730		324		
16⅔% Flake		7.740		324		
		7.680		321		
24	4	7.470	7.502	269	268.5	28
		7.540		272		
14.3% Flake		7.500		266		
		7.500		267		
27	4	7.375	7.400	235	238.0	31
		7.405		240		
12.9% Flake		7.420		239		
		7.400		238		
30	4	7.320	7.309	213	213.75	34.2
		7.305		215		
11.8% Flake		7.305		213		
		7.305		214		
34	4	7.200	7.240	191	192.75	37.6
		7.270		194		
10.5% Flake		7.240		194		
		7.250		192		
15	3	7.760	7.762	430	428.0	18.1
		7.780		427		
16⅔% Flake		7.720		426		
		7.780		429		

After 16 Runs. Per Cell	Total 2 Cell 1st low pate hot 2nd high " "	Total 4 cells.	After 50 Runs. or		
.2557	.5772	1.1884	.0061	.0199	.1437
.3215			.0138		
.3177	.6112		.0713	.1238	
.2935			.0525		
4.5515	9.1187	15.1752	1.0514	2.4320	3.8029
4.5672			1.3806		
3.2045	6.0565		.9152	1.3709	
2.8520			.4557		

Mg per Drump Hyd.	Flake	Loading Wt.	Average	No. of Drumps	Average	Mg per Drump.
27	3	7.330		241		
		7.305	7.314	245	241.75	30.2
10% Flake		7.305		241		
		7.325		241		
30	0	6.830		226		
		6.860	6842	228	226.0	30.2
		6.855		226		
		6.845		224		

	16th run Average	Average Sediment after 16 Run		3rd run Average		
20/4	1310	.1240	7.711	1241	322.0	24
24/4	1382	.1870	7.502	1295	268.5	28
27/4	1366	.2665	7.400	1286	238.0	31
30/4	1322	.2880	7.309	1261	213.75	34.2
34/4	1344	.3225	7.240	1244	192.75	37.6
15/3	1345	.1680	7.762	1262	428.0	18.1
27/3	1371	.2970	7.314	1283	241.75	30.2
30/0	26.	3.7950	6.842	62	226.0	30.2

Tubes from Cells A4

3374 Sept (?) 1909

32574 May (?) 1911

3374 Round bottom caps in 3374 tubes sawed with difficulty. Heated greatly and burnt up two saws in cutting 4 tubes. Single dump loading. Rings quite loose and slipped off with comparative ease.

3257 Nothing unusual.

MC Claim Expt

(1) Tube loaded with 1256, no flake

(2) Same quantity before loading.

(3) Same as 1 except reg. by chute.

(4) " " 2 " " "

To determine condition as to fines.

Also the segregation of fines in loading machine hopper is to be determined.

Tubes loaded with Hydrate.

1256	407	dumps.
1048	329	"

Material removed from 4 such tubes and carefully weighed by Dr Greenley as follows:

Wt per tube	1256	9.198	gm.
	1048	9.295	"

Net dumps after reaming not definitely known but probably:

1256	397	dumps.
1048	321	"

Weight per dump.

1256 $\frac{9.198}{397}$ = 23.2 mg.

1048 $\frac{9.295}{321}$ = 29.0 "

Loaded on machine #164
4/21/15. (over)

Specific gravity ~~was~~ of the two hydrates ~~after~~ before loading was

#1256	3.750	
1048	3.950	Ratio 1.053

Weight of equal volumes, dry as loaded,

1256	6.260 gm.	
1048	6.553 "	Ratio 1.047

Loading ratio (see previous page)

1256 per drop	23.2 mg.	
1048 " "	29.0 "	Ratio 1.250

The latter ratio is probably due to the more powdered form of 1048 which would therefore load more compactly in the small opening of the slider.

When water was poured on, an ebullition took place accompanied by a hissing sound, suggesting some form of chemical action.

Samples of old and new hydrate after loading without flake were placed in tall graduates of distilled water and allowed to settle.

1256 (old) settled rather much slowly than 104 S (new). When settled 1256 showed very distinct coarse particles, sharp and well defined, at the bottom. Above this a layer of finer particles and then, after a distinct line of demarcation, about $\frac{1}{8}$" of very fine powder. The total amount was about $1\frac{1}{2}$" to 2" in depth.

104 S after settling showed no distinct lines but varied gradually from a fine powder to a coarse powder. There were very few distinct particles as in 1256.

104 S occupied at least space when settled than did 1256, on account of greater average fineness.

When opening up the tubes loaded with out flake it was found that a layer of the active material readily flaked away from the pencil of material. This effect was equal approximately with both samples of hydrate.

When tubes sawed lengthwise material adjacent to tube was withdrawn by saw.

This latter effect has also been noted with tubes loaded with flake in regular way.

This is probably due to the edge not being compressed by tamping rod in loading.

Tubes loaded with
Flake Only

Required 5020 tamps and
1 hr. 20 min. to load.

Object to demonstrate
spring effect of flake
against expansion and
contraction of hydrate.

Experiment to demonstrate different bulk of old and new hydrate -

Tubes loaded in Arohem machine
" not perforated and without spiral seam. Simply a straight tube of metal. No flake used.

① Machine adjusted to load 24 mg. per dump. $Ni(OH)_2$ 102 Sp.
294 dumps.

② Adjustment not changed. Loading weight now 21 mg. per dump. $Ni(OH)_2$ #1256
323 dumps.

③ Machine adjusted to load 24 mg. per dump. $Ni(OH)_2$ #1256
Wt. 6.438 grams. 282 dumps.

④ Adjustment not changed. Loading weight now 26 mg. per dump. $Ni(OH)_2$ 102 Sp.
Wt. 6.718 grams. 294 dumps.

Material removed from 'unperforated tubes, mentioned on the next preceding page, and water poured over them. Gas (possibly occluded air) was driven off, followed by disintegration.

1256 separated into the distinct dump layers.

102 Sp. fell into an indistinct powdery mass.

Other samples were treated similarly, using Renewal Solution instead of water.

Both samples fell into powder.

Pettit v Reed R.R. 9269 R

A6 Cells 78873 - 78968

Cell filled with soft slimy sediment

Tubes soft — material squirts out when squeezed.

Tubes from each cell cut open and found to contain exceedingly thin layers of flake.

This bears out theory previously stated.

Cells to be retained in Repair Lab for possible future evidence.

479 N and 479 F

opened (April 1915) and sediment measured

479 N	4 grams
479 F	133 "

479 N has old Ni plates new Fe
479 F " old Fe " " Ni

Amounts of iron and nickel in each cell about equal.

This would seem to indicate that Fe comes out more when plates are old, while Ni comes out more when plates are new.

Robert Gair Co. RR. 10191 R.

Tubes empty - no hydrate or flake.

Probably no flake loaded in these tubes as it could not have come out through perforations.

Very soft tubes from same cells show lack of flake in soft parts.

Hydrate loaded in tubes without perforations: May 6, 1915.

1256 – Standard hydrate (no cobalt).
5635 – Special hydrate (recent mfr. – no cobalt
102 Sp. – Regular hydrate (with cobalt.

	Hydrate	Mg. per dump	No. of dumps	Weight per tube.
1	1256	26	280	6.886
2			286	6.990
3			289	6.873
4			282	6.915
5	5635	26	290	7.056
6			293	7.066
7			292	7.090
8			289	7.082
9	102 Sp.	26	291	7.082
10			289	7.115
11			292	7.090
12			289	7.082

opened up and material floated.
∴ allowed to settle in water.
1256 lighter color than 5635, which is lighter than 102 Sp.
Original particles in 1256 & 5635 – none in 102 Sp.

Archer says that originally Mr. Edison rigidly insisted upon flake not less than 4 mg. per dump. Later this low limit was reduced over Archer's protest to 3.4 mg. per dump.

Dr. Greenley says that on visit to factory in summer (June?) 1914, Mr. Edison looked over flake report which showed average of about 3.7 mg per dump. At first said this was too low. Then asked number of dumps per tube and was informed. He then did some figuring and was satisfied.

He must have had the amount per tube in mind and to have missed the importance of the higher weight per dump.

Curly flake will load heavier than flat flake.

Regular $\frac{1}{4}$" flake .062" square
$\frac{1}{8}$" flake .050" "

1/4" tamp

Main tamp	2 lb.	8 oz.	= 40 g.
Rebound	1 "	0 "	= 16 "
Total	3 lb.	8 oz.	= 56 g

Tooth pitch .435"
Lift 10 teeth

3/16" tamp

Main tamp	1 lb.	4 oz.	= 20 g.
Rebound	0 "	8 "	= 8 "
Total	1 lb.	12 oz.	= 28 g.

Tooth pitch .435"
Lift ?

Timing of Tamps in Tube Loading Machines

I timed one machine on 5/18/15 and counted 42 tamps per minute

Lystrup says standard is 41, but formerly on 4th floor in old building it was from 36 to 40

When loading employed "single dump per tamp" it took 17-18 minutes to load tube and average for tube ran about 340 or 680 tamps.

$\frac{680}{17.5} = 39$ tamps per minute

but only 20 double dumps per minute.

Lystrup may have meant "double dump" loading in which case there were still only 20 double dumps per minute!

With present rate there are 41 x 3 = 123 single dumps

(over)

or 62 double dumps per minute.

Archer loads at rate of 48 to 50 tamps or 25 double dumps per minute.

Speed of loading may be too high to prevent nicing. This should be investigated.

If speed is over 42 tamps per minute, green tops are caused because flake feed is cut off before hydrate feed. Probably green portion however is not more than 3 or 4 dumps or is later reamed out.

What should speed be to get 40 double dumps per minute?

$40 \times 2 = 80$ single dumps.

$\frac{80}{3} = 27$ tamps per minute.

Speed of 3/16" machines is 32 p. min.

These machines timed today (5/28/15) by me and found to run at rate of 35 tamps per minute

Special test - Jan. 3, 1911

weight per dump.

Flake	4.35 mg.
Hydrate	27.85 "

These weights were regular at that time.

Reaming per tube	.287 gm.
Waste " "	.399 "

Weight of flake per tube	1.436 gm.
" hydrate " "	9.144 "

Dumps per tube	320 -
Lines " "	293 -

Lystrup has detailed record.

5/27/15.

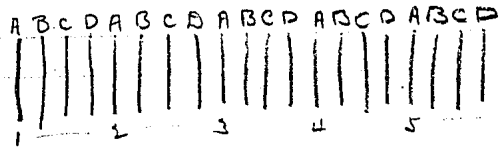
Experiment

J3 cell to be made to test effect of poor flake feed on bursting

Each plate to contain

A. 5 tubes Regular
B. 5 " 1/8" flake
C. 5 " Reg. flake with 3 ½" green sections
D. 5 " 1/8" " " " " "

arranged as follows:-



6/5/15. Short circuit developed on second charge.
First run 43 to 1.V. 48 to .5V.
Sh. Ci. due to distortion of tubes. All tubes twisted & buckled. Sl. tendency of screws to flare. Tubes straightened and cell reassembled.

B4 Cells from fire.

Material in parts of plates which were evidently hottest is projecting through perforations like spikes as though it had squirted out and then dried and hardened.

Sediment in cells is much more readily washed out with hot water than with cold.

These indicate effect of heat on the production of sediment.

12/5/16.

Mr. Bachman announced at the regular Tuesday conference today that it had been decided to open the flake fingers of the tube loading machines so that the weight limits per tump would be 4.0 and 4.4 mg. At present it is usually below 4 mg. The idea is that now the weights will be nearer the 4.4 point.

[THIS BOOK WAS USED IN BOTH DIRECTIONS. THE FOLLOWING PAGES WERE FILMED FROM THE BACK END FORWARD.]

Factors for Potassa

K_2O .04713

KOH .03613

K_2Cl_2 .06913

Iron

There is no pressure control on Fe platens.

Items for investigation

Time between mfr. and packing
" " " " testing
" " " " 5 gm loading
8 gm machine at S.L.
Ad. sample here for test ?